METHOD OF PRODUCING FATTY ACID ALKYL ESTERS

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The invention concerns a method of producing fatty acid alkyl esters by transesterification of triglycerides with an alcohol in the presence of a basic catalyst and is characterised by a combination of the following steps: (1) the triglyceride is mixed with the alcohol and catalyst and converted to form two fluid phases, namely, a crude ester phase and a glycerin phase; (2) the two fluid phases are separated; (3) the crude ester phase is divided into two portions (A) and (B); (4) portion (A) is purified, producing substantially pure fatty acid alkyl ester; (5) portion (B) is mixed with more triglyceride for transesterification, more alcohol and more catalyst and converted to form two further fluid phases, namely, a crude ester phase and a glycerin phase; steps (2) - (5) are then repeated.

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Method to the Herstelluna of Fettsäurealkvlestern the invention relates to a method to the preparation of fatty acid alkyl star by transesterification of triglycerides with an alcohol in presence of a basic catalyst.

Bottom transesterification is the conversion of triglycerides, i.e.

vegetable and animal fats and oil to understand with alcohols, like methanol, ethanol, Butanol and isopropanol, in particular methanol and ethanol, whereby the monoesters of the fatty acids as well as glycerol develop. Fatty acid methyl esters win more as Diesel replacement material ever at importance.

The transesterification begins with a Zweiphasensystem from triglyceride and alcohol, which contain the catalyst, whereby however with increased reaction progress and formation of ester an homogeneous phase develop, which becomes again biphasic by formation and excretion of glycerol (crude ester phase and Glycerinphase).

From at-B 394,374 a such method is known. In accordance with this method an excess of the used alcohol from 1,10 becomes to 1.80 mol for each mol with glycerol esterified fatty acid used. From at-B 388,743 a method is out < to the preparation of a fatty acid mixture; RTI ID=1.1> Abfallfetten< /RTI> and/or. oil and the use of this mixture as force and/or. Fuel known.

Also at-B 397,966 describes the preparation of fatty acid esters of low monohydric alcohols by transesterification of for example rapeseed oils. In accordance with this method the transesterification in presence of a solid basic catalyst is < with one; RTI ID=1.2> Katalysatorüberschuss< /RTI> of at the most 1.6 mol for each mol as Glycerid bonded fatty acid conducted.

From at-B 397,510 is a two and/or. multistage transesterification procedure known.

As catalysts for the transesterification usually basic catalysts become, e.g. Alkali hydroxides, metal hydrides, - alcoholates, - carbonates or - acetates, and acidic catalysts, e.g. Mineral acids, used. The most frequently used catalysts are sodium and potassium hydroxide as well as Natriummethylat, which becomes e.g. admixed in alcohol dissolved a vegetable oil. A such method is from at-B 386,222 known.

Prior transesterification procedures, which become conducted with room temperature, need at least 2 transesterification stages, in order to increase the yield at esters on over 90%. With an alcohol employment of < RTI ID=2.1> 130-150%< /RTI> the stoichiometric necessary amount and a cousin no set of 1,2-1,5 weight percentage of the used triglyceride an yield of 80-90% achieved becomes in the first transesterification stage. After separations of the Glycerinphase after-esterified becomes in a second Umesterungstufe, whereby the yield on 99-99,3% increased becomes.

With this previously known method the entire alcohol/catalyst quantity in the ratio becomes 75:25 on both steps divided, so that a relative high excess is present in each case.

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The invention places itself the object to place an improved process to the preparation of fatty acid alkyl star by transesterification from triglycerides to the order is other reduced with which Alkoholund catalyst quantity.

The invention process to the preparation of fatty acid alkyl star by transesterification of triglycerides with an alcohol in presence or several basic catalysts, is characterized by the combination of the subsequent steps that (1) the triglyceride with the alcohol < and (); RTI ID=2.2> Catalyst (EN) < /RTI> mixed and bottom formation of two liquid phases, a crude ester phase and one
Glycerinphase, reacted becomes; (2) the two liquid phases from each other separated become; (3) the crude ester phase into two parts (A) and (B) divided becomes; (4) the part (A) of the crude ester phase purified becomes, whereby essentially pure fatty acid alkyl ester becomes obtained; ; (5) the part (B) with other, umzuesterndem triglyceride, other alcohol and other (n) < RTI ID=3.1> Catalyst (EN) < /RTI> mixed and bottom formation of two other liquid phases, an other crude ester phase and an other Glycerinphase, reacted becomes; according to which the steps (2) to (5) repeated become.

In the invention process also a mixture of catalysts can become used.

A prefered embodiment of the invention process consists of the fact that the crude ester phase in the step (3) in the ratio between 1:1 and 1:99, in particular between 1:3 and 1:99, becomes in part (A) and part (B) divided.

The purification of the crude ester phase becomes best by water washing, in particular hot water, made.

The invention process can become continuous conducted.

As alcohol for the transesterification according to invention methanol or ethanol particularly good is suitable.

The invention process is characterised furthermore by it that it can become bottom standard conditions, thus performed with room temperature and normal pressure.

As triglycerides fats can and oil vegetable and animal origin used to become in the invention process.

Mentioned become exemplary: Corn germ oil, cottonseed oil, soya oil, sunflower oil, rapeseed oil, peanut oil, Kokos and other palm oils, flax seed oil, tallow tree oil, Rhizinusöl, fish oil, Waltran, tallow, Schweienfett, oils of sea and land animals, oils from bacteria, algae as well as vegetable lipids.

It has itself shown that the invention process an other reduction of the alcohol and catalyst need allowed, whereby the conversion degree over 99% amounts to.

By the cycle guidance of the part (B) of the crude ester phase the alcohol and catalyst portion of recycled contained in this phase become, so that can become lowered by this enrichment the need at other alcohol and catalyst. This has the advantage that can become lowered when starting the invention process the entire alcohol quantity on approximately 125% of the stoichiometric necessary amount. The catalyst quantity can become on 1,1% the Triglyceridmenge lowered.

It has itself furthermore shown that the required alcohol quantity can become even so far lowered that no alcohol recuperation from the crude ester phase is necessary.

The procedure guidance according to invention can on dynamic control and/or. measurements of volume in each phase, as well as without filling volume measurement do. With continuous driving fashion the four process parameters become, i.e. Supply of triglyceride, alcohol/catalyst mixture and part (B) of the crude ester phase, as well as discharge of part (A) of the crude ester phase, fixed adjusted, so that the invention process without other monitoring can run.

The optimization of the invention process can become simple over the adjustment of the ratio of part (A) part (B) of the crude ester phase made. The need at rule and measuring technique becomes practical reduced on zero.

The instant invention becomes subsequent described still near in a preferable embodiment, whereby in the example A those Preparation of the starting load in accordance with the known two-stage transesterification described will and in the example B the invention process described becomes.

Example A a transesterification reactor equipped with stirring means became with 1.000 kg vegetable oil (filtered; Moisture content: < 0.1 Gew. - %) charged. Subsequent one became bottom agitations a mixture of 120 kg methanol and 10.5 kg KOH added and after end of the addition still 30 minutes agitated.

Afterwards the reaction mixture became pumped into a settling tank, in order to let the phases train. After formation of the phases the Glycerinphase became separated and the remaining phase, which were a mixture of oil and ester, into the reactor back-pumped, where them became again with a mixture of 40 kg methanol and 3.5 kg KOH the conversion brought.

Subsequent one was let train the reaction mixture recently into the settling tank pumped and the phases. The ester phase became after separations of the Glycerinphase with water up to the neutral point washed and a white ester content of 99,4 Gew. - % up.

Example B it became a first mixture from methanol and KOH prepared, with ester, which became in the example A obtained, in the weight ratio 1: 9 (Methanol/KOH + triglyceride: Esters) mixed and the transesterification reactor supplied. The transesterification became made with ambient temperature in a time of approximately 25 minutes.

After the transesterification the formed phase mixture the reactor became removed and in a centrifuge in Glycerinphase and ester phase separated.

10 < RTI ID=6.1> Gew. - 8< /RTI> the obtained ester phase became as part (A) into a container given, and after a rest time of approximately 2.5 hours the decanted was < RTI ID=6.2> Übrestand< /RTI> with hot water washed, in order to remove catalyst, methanol, soaping Glycerinreste. Still the Glycerinphase separated by gravity became down withdrawn. Afterwards the wash water became separated by centrifugation. The obtained esters was ready for use and did not need not other purified to become.

The remaining 90% of the obtained ester phase became as part (B) for a next transesterification used and with a second mixture from methanol and KOH in the weight ratio 1: 9 (Methanol/KOH + triglyceride: Esters) mixed and the transesterification reactor supplied. The other procedure way was already above described like.

In accordance with the described above embodiment thus in each case 10 Gew became. - % of the obtained ester phase from the method removed and 90 Gew. - % of the obtained ester phase for the next transesterification recycled. Same good results can become obtained, if between 1 and 25 < RTI ID=6.3> Gew. - < /RTI> Ester phase recycled become, if between 99 and 75 Gew. - Become % ester phase recycled.

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Claims:

1. Method to the preparation of fatty acid alkyl star through
Transesterification of triglycerides with an alcohol in presence or several basic catalysts, characterized by the combination of the subsequent steps that (1) the triglyceride with the alcohol and ()
Catalyst (EN) mixed and bottom formation of two liquid phases, a crude ester phase and one
Glycerinphase, reacted becomes; (2) the two liquid phases from each other separated become; (3) the crude ester phase into two parts (A) and (B) divided becomes; (4) the part (A) of the crude ester phase purified becomes, whereby essentially pure fatty acid alkyl ester becomes obtained; ; (5) the part (B) with other, umzuesterndem triglyceride, other alcohol and other (n) catalyst (EN) mixed and bottom formation of two other liquid
Phases, an other crude ester phase and an other
Glycerinphase, reacted becomes; according to which the steps (2) to (5) repeated become.

2. Process according to claim 1, characterised in that those
Crude ester phase in the step (3) in the ratio between 1:1 and
1:99 in part (A) and part (B) divided becomes.

3. Process according to claim 2, characterised in that those
Division in the ratio between 1:3 and 1:99 in part (A) and
Part (B) made.

4. Process according to one of claims 1 to 3, characterised in that the purification of the crude ester phase through
Water washing made becomes.

5. Process according to one of claims 1 to 4, characterised in that it continuous conducted becomes.

6. Method after or the several claims a 1 to 5, characterised in that as alcohol for the transesterification
Methanol or ethanol used becomes.

7. Process according to one of claims 1 to 6, characterised in that it with room temperature conducted becomes.

8. Process according to one of claims 1 to 6, characterised in that a catalyst or a mixture of
Catalysts used become.

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